# Research Brief for DOE/IHEA Process Heating Materials Forum

Research Title: Surface Modification and Thermomechanical Properties of Refractory and Insulation Materials

### **Industry Need:**

The process heat industry has a need for improved life of insulation and refractory materials used in the areas of heat generation, heat containment, waste heat recovery, and materials handling. Improvements in performance coupled with a better understanding of the behavior of these materials could lead to reduced operating and materials costs, reduced process down time, and improved equipment life. The proposed research would examine the surface modification of insulation materials and development of unique characterization techniques.

#### **Existing Research:**

Currently, Oak Ridge National Laboratory (ORNL) has a project in collaboration with the University of Missouri-Rolla (UMR) and several industrial partners on the surface modification of refractories that would be in direct contact with molten metals and glass. The project is using high density infrared (HDI) treatments to reduce surface porosity, allow surface chemistry changes to be performed, and improve mechanical properties. To date, HDI treatments have fused the surface regions at moderate power levels (≥1375 watts/cm²). During solidification of the surface melted region on alumino-silicate materials, mullite grains form that are highly oriented with the c-axis perpendicular to the surface.¹

Surveys have been performed in the past of the various methods for measuring thermal conductivity/diffusivity of refractory materials. One recent study performed at ORNL with collaboration from Orton Ceramic Foundation compared results for three common ASTM methods (calorimetry, hot-wire, and laser flash) using a commonly available fine-grained alumina material<sup>2</sup>. This work showed how results from these methods can vary widely for a single material depending on sample size and microstructure, causing difficulty in obtaining a value of confidence for a material and discrepancy when comparing thermal conductivity Further, it was shown that a non-point-source values obtained by different methods. measurement method is needed for refractory materials, which incorporates the rapid measurement of the laser flash technique, while allowing for the large sample sizes used in the calorimetry and hot-wire techniques. ORNL, in collaboration with the UMR, has extensive experience in the thermomechanical testing of refractory materials for the glass industry. Physical (density, porosity, chemical composition), thermal (conductivity, expansion, spall resistance) and mechanical (creep, modulus, MOR, strength) property data along with microstructures for various competing brands of refractory materials have been studied to date. Classes of materials which have been studied include conventional silica, high purity MgO, fusion-cast AZS, mullite, and fusion-cast alumina.

<sup>&</sup>lt;sup>1</sup> T. N. Tiegs, J. O. Kiggans, F. C. Montgomery, C. A. Blue, and M. Velez, "High Density Infrared Surface Treatment of Ceramics," to be published in Ceram. Trans., Am. Ceram. Soc., Westerville, OH (2003).

<sup>2</sup> I.G. Henrick, C.W. Kistler, Ir. A.A. Wereszczak, and M.K. Ferber, "Thermal Conductivity of Alumina.

<sup>&</sup>lt;sup>2</sup> J.G. Hemrick, C.W. Kistler Jr., A.A. Wereszczak, and M.K. Ferber, "Thermal Conductivity of Alumina Measured with Three Techniques" submitted to Journal of Testing and Evaluation (August 2002).

## **Proposed Activity:**

The proposed research would examine the surface modification of insulation materials and development of unique characterization techniques. Both approaches utilize the high density infrared (HDI) source at ORNL. This unique instrument is capable of generating extremely high power densities on the order of 3.5 kW/cm² using a single lamp. HDI is capable of heating the near-surface region of materials to very high temperatures where sintering, diffusion, and melting can occur.

The surface modification portion of the research would examine use of the HDI system to densify the surface of insulation and other porous refractories. Refractory insulation is designed to be porous, however the porosity also allows corrosive species to migrate into the insulation resulting in degradation of the insulation itself or the backing material. Both would reduce the lifetime of the structure. By treating just the surface region, the refractory could be sealed to minimize degradation, while also maintaining the insulating properties of the original material.

It is also proposed to develop a new method for determining thermal conductivity which is capable of measuring heterogeneous, large grained materials in a brick geometry over a temperature range of room temperature up to 2000°C. This will be accomplished using the HDI lamp which is ideal for heating a material to high temperatures along with providing concentrated heating to a sample in very short periods of time. Therefore, using a single instrument, a refractory sample could be heated to a specified test temperature and then tested at temperature by "flashing" the brick surface in a fashion similar to the laser flash technique. Yet, as opposed to the small spot area (≈10mm) sampled by the laser flash technique, a large surface area can be heated using the IR source. An array of temperature sensors on the back surface of the refractory will be used to measure the temperature increase due to the thermal flashing and the subsequent heat flow through the refractory brick. Thermophysical characterization of competing brands and types of refractory and insulation materials for the Process Heating Industry will be evaluated using the method developed above for measuring thermal conductivity/diffusivity, along with the presently existing equipment at ORNL for measuring density/porosity, chemical composition, thermal expansion, creep, modulus, strength and microstructure. Results can then be used by the industry for improved process design and better materials selection.

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